

(19) FRENCH REPUBLIC

NATIONAL INSTITUTE OF  
INDUSTRIAL PROPERTY

PARIS

(11) Publication No.  
(to be used only for copy requests)

(21) National Registration No.

2 630 927

88 06192

(51) Int. Cl.<sup>4</sup>: B 01 L 3/00; B 65 D  
47/00

(12)

PATENT APPLICATION

A1

(22) Filing Date: May 3, 1988

(30) Priority:

(43) Date of laying open of the  
application to the public:  
"Patents" OIPB No. 45 of November  
1998.

(60) References to other related  
national documents:

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(54) Container Head for Chemical Reaction, Treatment or Analysis of Various  
Products, and Container Using the Same

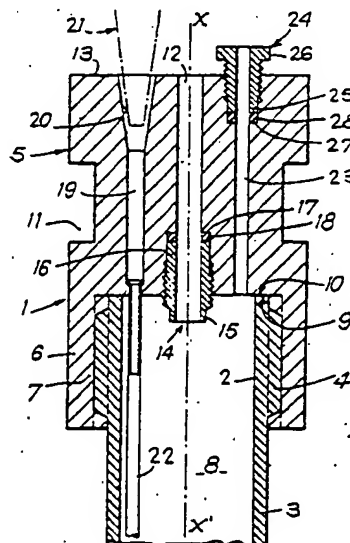
(57) Chemical industry.

The head comprises a body forming  
an annular skirt 6 which is provided  
with means for the hermetic mounting  
thereof on a neck 2 of a container 3,  
and which extends from a base 10 as an  
adapter part 5 through which pass:

- at least one injection/sampling  
conduit 19, which is extended by a dip  
tube 22; and

- at least one through passage 23  
provided with an accessory fitting  
connector 24 at the upper face of the  
head.

Use in small-volume containers  
for chemical analysis.



CONTAINER HEAD FOR CHEMICAL REACTION, TREATMENT OR ANALYSIS OF  
VARIOUS PRODUCTS AND CONTAINER USING THE SAME

The present invention relates to containers which are primarily used in the fields of the chemical and physical chemistry industries, in the treatment, analysis or reaction of samples of products which have been developed or are under development as part of industrial processes.

Container heads are well known in the prior art. The containers are usually made of glass, with flat or rounded bottoms, and have diameters and heights which vary greatly depending on what they contain.

In development of a multi-stage production process which requires ten, fifteen, or even more steps between primary materials and finished product, the complexity of the chemicals studied and the difficulty involved in producing them make it necessary to work with small quantities of the product in small-volume containers of the order of 1 to 15 ml.

At the present time, largely due to advances in analysis techniques, it is possible to work with very small quantities, of the order of a few milligrams. The container which contains the reaction material must, however, allow all normally required operations to be performed, including sampling, addition of products, reflux, inert gas injection, agitation, etc.

Such containers commonly have to be closed, at least temporarily, and for this reason are provided with a smooth neck or a neck shaped to so as to allow a stopper to be fitted.

Known and recommended methods for ensuring hermetic fitting of a stopper in the neck are common in design and generally exploit an elastic reaction of at least part of the stopper to achieve a hermetic seal at a relative pressure which forms in the container, or a relative vacuum maintained therein. Known stoppers may be either closed or open, and in such cases they usually comprise a septum.

Known stoppers can be considered satisfactory in terms of closing or hermetically sealing containers.

These stoppers are not, however, suited to the requirements for usage or handling of small-quantity containers, and particularly for proper performance of operations involving heating or cooling of a product, temperature measurement, temperature regulation by means of a sensor, partial sampling of the product or compound, or adding of an additive or reactant.

In order to perform these operations well, it is generally necessary to remove the stopper, except in cases where this is of the type having a septum, where the operations to be carried out can be performed by means of a needle type injection/extraction apparatus.

In all other cases, it is necessary to remove the stopper, with all the risks that accompany this, such as contamination of the sample; pollution of the environment; deterioration of the stopper; and rupture, breaking or deterioration of the container, which results in spilling of the product contained therein.

The present invention is intended to solve the problems described above by means of a new container head designed so as to allow for rapid and practical fitting on a container, while allowing, after this fitting, for the possibility of various operations on, or measurements of, the sample contained therein, without this having to be removed from the container.

The container head according to the invention is also conceived in a manner that allows it to be used as a closing device, regardless of the operations to which the sample is to undergo thereafter, whether this process does not involve any intermediate steps or whether this requires multiple operations such as injection, extraction, reflux, agitation or heating, which must be performed on the sample, from the exterior and without opening the head.

In order to solve these problems, the invention recommends forming a container head so that it comprises an annular skirt provided with means for hermetically mounting it on the neck of a container, and so that it extends from a base as an adapter head, through which pass:

- at least one injection/extraction conduit, extended by a dip tube;
- at least one through passage provided with an accessory fitting connector at the upper face of the head.

Various other characteristics are made clear by the description hereinafter with reference to the appended drawings which illustrate as non-limiting examples, modes of embodiment of the object of the invention.

FIG. 1 is a sectional elevation illustrating the object of the invention.

FIG. 2 is a sectional elevation illustrating an example of application of the stopper [sic].

FIG. 3 is a schematic perspective view showing a variation on the embodiment of an element of the object of the invention.

FIG. 4 is an exploded perspective view showing an advanced embodiment of the stopper.

FIGS. 5 and 6 are partial sectional elevations showing two variations on the embodiment of one of the constituent elements of the object of the invention.

In the mode of embodiment according to FIG. 1, the head according to the invention, which is indicated in its entirety by reference numeral 1, is intended for mounting on a neck 2 of a container 3 which is made, for example, of glass. The container 3

is not an integral part of the invention and can therefore have any configuration suitable to the field of application in question, being treatment, analysis or reaction of products or compounds. In this respect, the neck 2 can be smooth or can comprise fitting means 4 such as threading or a sealing flange.

The head 1 according to the invention is made of any suitable material, rigid or elastically deformable, such as polytetrafluoroethylene, preferably by molding. The head 1 comprises an upper section 5, which extends, from the base thereof, as skirt 6, the inner peripheral surface thereof comprising a means 7 for interaction with the means 4. In this case, the means 7 take the form of a thread intended to interact with the complementary thread which constitutes means 4. The head 1 is intended to create a hermetic seal capable of maintaining a relative pressure or vacuum between the external environment and a confined interior space 8 of the container 3. The hermetic seal is, for example, created by cooperation between a planar annular transversal rim 9 of the neck 2 and an abutment 10 which delimits the skirt 6, this abutment 10 can also be termed the base of the section 5. Packing can also be installed, for this purpose, in a groove provided at the abutment 10, or sandwiched between the rim 9 and the abutment 10.

The upper section 5 may have various external configurations, depending on whether manual or automatic assembly or disassembly is intended. Gripping means for these purposes are not described in particular hereinafter, as they are well known in the art based on known products. Section 5 preferably comprises a peripheral annular groove 11 of a height and depth determined according to the dimensional characteristics of hands or of gripping tongs on the manipulators of treatment, analysis or reaction equipment. The groove 11 can be complemented by or replaced by bands.

According to the invention, the upper section 5 comprises a hole 12 which is a through hole, that is to say, opening in both the base 10 and a top surface 13 of the section 5. The hole 12 is preferably, but not necessarily, provided on the axis of rotation  $x - x^1$  of the head. The hole 12 has a constant diameter over its entire length, which corresponds to the height of the section 5. The hole 12 can be formed by any appropriate means so as to be provided with, preferably, a stuffing-box 14, comprising a tubular sleeve 15 which can be screwed into a threaded hole 16, the end thereof forming an abutment for pressing on packing 18.

The section 5 also comprises an injection/extraction conduit 19, preferably parallel to the axis  $x - x^1$ , which is also provided in a through manner so as to open on the base 10 and the top 13. Beginning from the top 13, the conduit 19 comprises a truncated conical section 20, the apex thereof being connected to

conduit 19, while the base thereof is situated in the plane of top 13. The truncated conical section 20 is intended to allow for the fitting of a tip 21, having a complementary shape, which can be either a stopcock or the appendage of an injection/extraction device. The conduit 19 is complemented by a dip tube 22 which extends beyond the base 10, to the interior of the container 3. Preferably, the dip tube 22 is of a length determined in accordance with that of the container 3, so that the extremity thereof opens in the vicinity of the bottom of such a container.

The section 5 is also provided with at least one passage 23, preferably parallel to the axis  $x - x^1$ , which is also a through passage which opens on the base 10 and the top 13. The through passage 23 is provided with, at top 13, a connector 24 which allows an accessory, which needs to be brought into contact with the internal milieu of the container 3, to be fitted. The connector 24 comprises a treaded tubular tip 25, combined with means for manual or mechanical gripping 26, which allow the tip 25 to be screwed into a complementary housing 27 provided, at the surface 13, on the axis of passage 23. The tip 25 is intended to squeeze a seal 28 against the bottom of the housing 27.

FIG. 2 shows an example of application of the head, equipped so as to be provided with a pipe 30, which dips into the interior of the working material, and which is provided in the hole 12, which holds it in a hermetic seal by means of stuffing box 14. The pipe 30 is preferably provided with two coaxial communicating routes 30a and 30b, so as to form an entrance and an exit route for a heat exchange liquid, supplied, for example, by a circulator device 31. The device 31 can be of any suitable type, and the representation thereof as a coil is only provided as one illustration thereof. The connection between the pipe 30 and the device 31 is accomplished by means of two tubes, 32a and 32b, which are formed by that section of the pipe 30 located outside the head 1. The tubes 32a and 32b can be axially separated as shown in FIG. 2, or concentric, as shown by FIG. 3.

The passage 23 can be occupied by a temperature measurement probe 33 which is hermetically fixed in place by mounting it in the connector 24 and screwing in the tip 25. The probe can be electrically connected, by means of a circuit 34, to a device for comparative measurement and analysis, with respect, for example, to a predetermined value, and capable of controlling either the temperature or the rate of circulation of the heat exchange liquid which circulates within the pipe 30.

The conduit 19 can be occupied by the tip 21, which is either a stopcock or an appendage of an extraction/injection apparatus.

The device 31 and the route 34 can be flexible, so as to allow for the movement of a container 3, closed by the head

according to the invention, from one work station to another, without the need to disconnect the device 31 and the route 34 from the container head 1. This mode of embodiment is particularly advantageous for use with automatic handling means.

A product, such as P, which is confined in the interior space 8 of the container 3 can, thereby, be subjected to treatment, analysis, or reactions, either automatically or not, by way of extraction or injection of additional compounds, and increases or decreases in temperature, based on regulation by means of the pipe 30, and monitoring by means of the probe 33.

Mounting of a drive shaft for an agitator, located in the container 3, in the hole 12, can be envisioned.

The means according to the invention allow for the container 3 to be hermetically sealed in a manner known in the art, while allowing for operations on the product P, when this is desired or is part of the treatment, analysis or reactions to be performed.

FIG. 4 illustrates the further possibility of providing the section 5 with passages 23a and 23b, which are also provided with connectors 24a and 24b at top 13, allowing for the connection of tubing by which products, such as gaseous products, can be introduced and/or recovered, where the supply thereof is necessary in the course of the treatment, analysis or reaction process, or where these form as the result of such a process. For example, flushing the portion of the interior space 8 which is not occupied by the product P can be envisioned.

FIG. 5 shows a variant embodiment wherein the conduit 19 opens in the center of the far end of a housing 40 which is provided at the top 13. The housing 40 is provided with a thread for screwing in a tubular connector 41 which forms, starting at a gripping means 42, a truncated conical section 20, provided for fitting of the tip 21. The connector 41 also serves to press an airtight washer 43 against a flange 22a of the dip tube 22, which is pressed against the far end of the housing 40.

FIG. 6 illustrates a second variant which consists of forming the section 20 as described above and providing, instead of a hole 19, a threaded housing 44, starting from the base 10. The housing 44 is intended to receive the flange 22a of the dip tube 22, which is pressed against the far end of the housing, with or without packing, by a threaded tubular connector 45 through which the dip tube passes and which is screwed into the housing 44.

These two modes of embodiment allow for the use of polytetrafluoroethylene dip tubes, and provide the most reliably hermetic seals.

In cases where at least one of the connectors 24 is not needed, it is possible to ensure hermetic sealing thereof by

replacing the tip 25 by a similar threaded plug. Similar means can also be used for the holes or conduits 12, 19 and 23.

The invention is not limited to the examples described and illustrated herein, as various modifications can be made thereto without going beyond the scope of the invention.

**CLAIMS:**

1. A container head for chemical reaction, treatment or analysis of various products,

characterized in that this comprises a body forming an annular skirt (6) provided with means for hermetic mounting thereof on a neck (2) of a container (3), which extends from a base (10) as an adapter section (5) through which pass:

- at least one injection/extraction conduit (19) extended by a dip tube (22); and

- at least one through passage (23) provided, at the upper face of the head, with a connector

(24) for mounting an accessory.

2. A container head according to claim 1, characterized in that this further comprises at least one hole (12) provided with a stuffing-box (14).

3. A container head according to claim 1, characterized in that the injection/extraction conduit (19) opens on the an upper surface (13) of the section (5) via a truncated conical section (20) for inserting a tubular tip (21) for closing this or for connecting an injection/extraction apparatus.

4. A container head according to claim 2, characterized in that the stuffing-box (14) is fitted into a housing (16) which is provided concentrically with hole (12) starting from the base (10) of the head.

5. A container head according to claim 1 or claim 4, characterized in that the hole (12) is provided on the axis of the head, and is occupied by the rotating shaft of an agitator.

6. A container head according to claim 1 or claim 4, characterized in that the hole (12) is occupied by a pipe (30) comprising two axially concentric and communicating routes which are extended outside the head as two tubes (32a and 32b) for connection to a device (31) for circulation of a heat exchange liquid.

7. A container head according to claim 1, characterized in that the section (5) possesses at least one through passage (23) which extends as a housing, threaded from the far end thereof, capable of receiving a complementary threaded tubular connector (25), and provided with a seal (28).

8. A container head according to claim 7, characterized in that the head (5) [sic] comprises at least one passage (23) occupied by a temperature measurement probe (33).

9. A container head according to claim 1, characterized in that the head (5) [sic] comprises an exterior peripheral gripping grove (11).

10. A container for treatment, analysis or reaction of various products comprising a head according to one of claims 1



to 9.

11. Use of the container head according to one of claims 1 to 8 in association with a device (31) and a route (34) which are flexible.

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Fig. 1

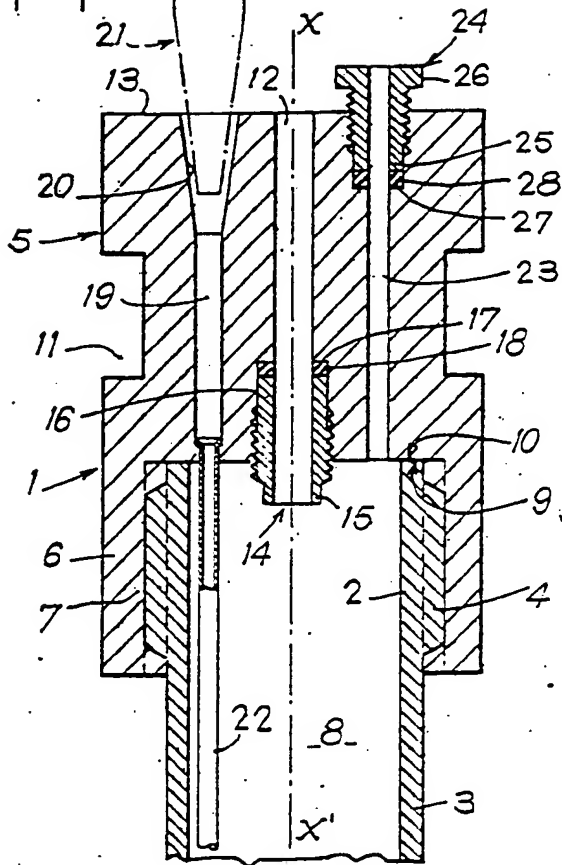


Fig. 2

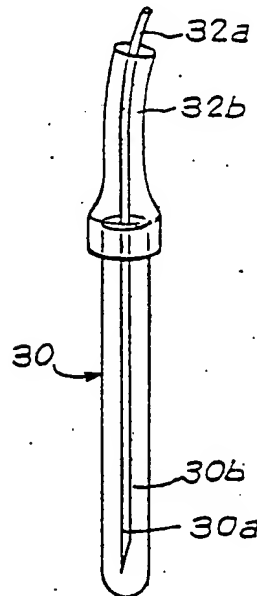
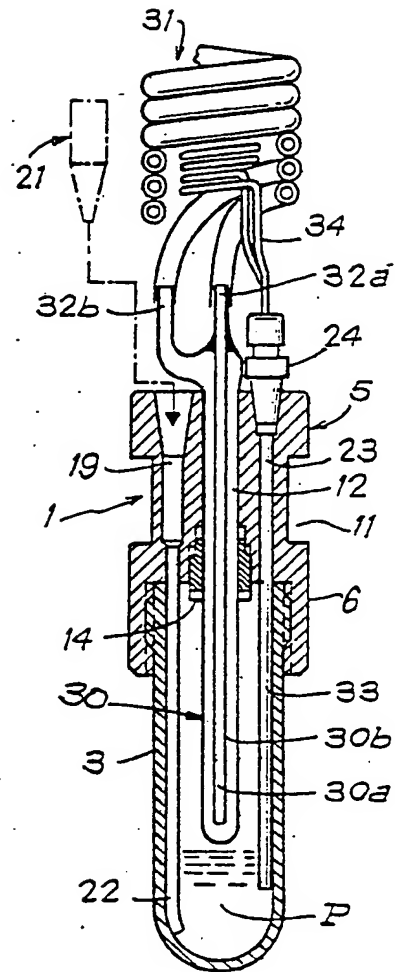


Fig. 3

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Fig. 4

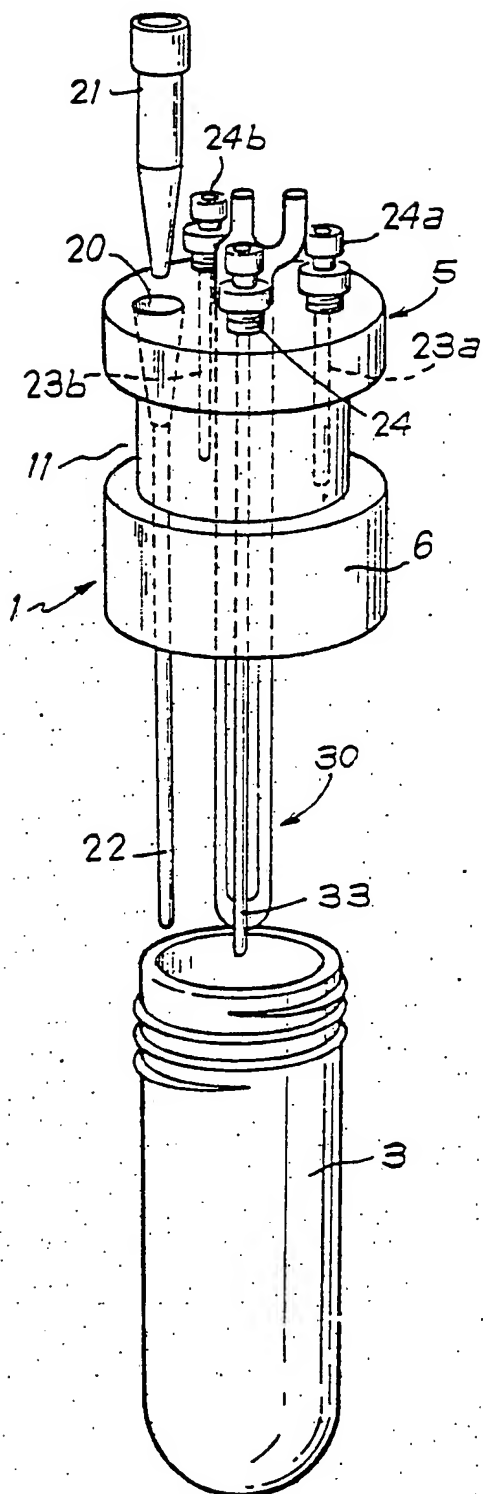


Fig. 5

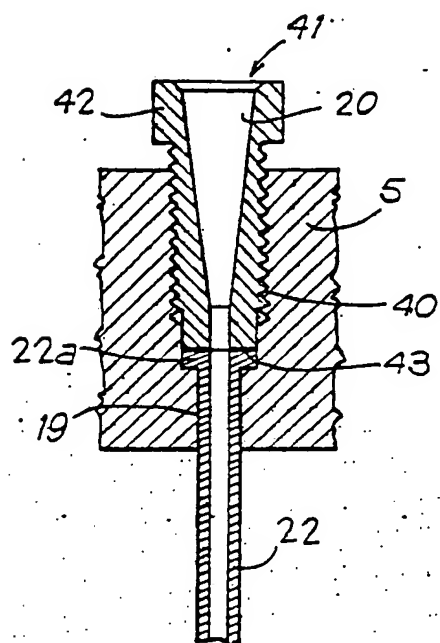


Fig. 6

